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KNOLLS ATOMIC POWER LABORATORY

SCHENECTADY, NEW YORK

AEC RESEARCH AND DEVELOPMENT REPORT

ELECTRODEPOSITION OF NICKEL ON URANIUM

A. P. Beard and D. D. Crooks

August 31, 1954

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Metallurgy and Ceramics

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DATE 12-2-55
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ABSTRACT

The application of electrodeposited nickel coatings, designed to protect uranium from destructive corrosion in boiling water, has been investigated in this laboratory.

Correlation between the pretreatment of the uranium and subsequent protection by thin nickel coatings has been established. Thin electrodeposited nickel coatings provide the best protection when applied to a matte surface produced by blasting with an aqueous suspension of silica (100 mesh) followed by a cathodic treatment in 35 wt % sulfuric acid, rather than to the rough surfaces produced on uranium by anodic pretreatments and acid pickling.

Blistering of nickel electrodeposits arising from hydrogen was encountered and eliminated.

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ELECTRODEPOSITION OF NICKEL ON URANIUM

A. P. Beard and D. D. Crooks

INTRODUCTION

The electrodeposition of metallic coatings on uranium involves problems similar to those encountered with other electropositive metals such as aluminum, magnesium, zirconium, etc. The ever-present oxide layer on the uranium[1] prevents as-plated metal-to-metal bonding. However, some approaches to achieve adhesion of electroplated metal to uranium are (1) by an immersion technique which replaces some soluble uranium compound on the surface with a continuous metal film on which metal is then electroplated, (2) by microscopic mechanical interlocking of an etched uranium surface with the electrodeposited metal, (3) by diffusion bonding of an electrodeposited metal to a uranium surface free of gross amounts of oxide.

In the investigation undertaken at KAPL, the objective included, however, not only the problem of adhesion of an electrodeposited coating on uranium, but also one of minimum porosity, since the oxidation of uranium by water and gases at elevated temperatures is rapid and destructive in nature.

The ultimate use of any coating technique developed in this work was on Hanford fuel slugs as a secondary protection for the uranium, as well as a bonding agent and a diffusion barrier between the uranium and the aluminum containers.

PRELIMINARY WORK

Earlier work on electroplating uranium with various metals has been reported by other investigators.[2,3,4] The coatings themselves, however, were not satisfactory for protection against hot or boiling water.

Using the reported plated procedures, coatings of copper, nickel, chromium, zinc, tin and composite coatings of these metals were tried on disks machined from transverse slices of a uranium rod which had been rolled in the alpha phase. However, very little progress was made in obtaining pore-free coatings on the flat surfaces of the disks that would withstand a boiling water test, although heat-treated copper-nickel composites showed some promise. In the earlier work an anodic treatment in 50 wt % trichloroacetic acid, followed by a nitric acid pickle and rinse was used as the first pretreatment. Examinations of surfaces and electrodeposits on them indicated that both were extremely rough. It was believed that elimination of the rough etch was necessary if pore-free coatings were to be obtained.

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Immersion plates of copper, zinc, and nickel were tried but resulted in little or no success. Silver, however, was deposited on uranium from solutions of silver nitrate and silver sulfate. The latter seemed to produce the best immersion coat on uranium but was not investigated further because of its high absorption cross section for thermal neutrons. The deposition of nickel coatings by reduction of nickel ions with hypophosphite[5,6] ions on the surface of uranium was also tried but was not successful in producing adherent, pore-free coatings.

EXPERIMENTS WITH ANODIC PRETREATMENT

Since little progress was made with pretreatment using trichloroacetic acid, immersion coatings, and composite electroplates, efforts were concentrated on finding a pretreatment for uranium that would permit electrodeposition of an adherent material that was corrosion resistant to water. On the suggestion of J. G. Beach (Battelle Memorial Institute), anodic treatments of uranium in phosphoric acid - water - hydrochloric acid solutions were tired. An etch that was considered satisfactory was obtained in a bath of the following composition and conditions:

H_3PO_4	50 vol %
H_2O	50 vol %
NH_4Cl	3.3 g/liter
Temperature	55°C
Current density	12.4 amp/dm^2
Time	10 min
Cathodes	graphite

The anodic treatment was followed by a nitric acid pickle and water rinse before plating.

Nickel was chosen as the electrodeposit to use in the development work because of (1) its excellent corrosion resistance in hot water, (2) the good throwing power of its electroplating baths, (3) the variety of electroplates with different properties that can be obtained, (4) the wide operating ranges and ease of control of the baths in general, and (5) the report by previous investigators[3] that nickel seemed to provide good protection for uranium in air.

Uranium, given the new pretreatment and plated with nickel from a Watt's bath, was found to be protected more than when using the trichloroacetic acid pretreatment, but the plates were still porous. Experiments showed that the uranium reacted with the Watt's nickel bath but not, however, with

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a "High Sulfate" bath consisting of nickel sulfate, sodium sulfate, boric acid, and ammonium chloride. The latter bath provided a satisfactory strike on uranium and allowed build-up of the nickel deposit to desired thickness in the Watt's bath.

A series of uranium disks was prepared in the following manner:

1. Vapor degrease.
2. Anodic treatment in phosphoric acid - NH_4Cl - H_2O solution.
3. Rinse.
4. Pickle in 50 vol % HNO_3 at room temperature.
5. Rinse.
6. Strike from "High Sulfate" nickel bath:

$NiSO_4$ $6H_2O$	70 g/liter
Na_2SO_4 $10H_2O$	160 g/liter
H_3BO_3	15 g/liter
NH_4Cl	7 g/liter
Current density	2.7 amp/dm^2 (25 asf)
pH	5.5
Temperature	40-45°C
Time	10 min

7. Rinse
8. Build-up of nickel thickness in Watt's bath:[7]

$NiSO_4$ $6H_2O$	325 g/liter
$NiCl_2$ $6H_2O$	45 g/liter
H_3BO_3	30 g/liter
Current density	4.9 amp/dm^2 (45 asf)
pH	4-4.5
Temperature	55°C
Time	~20 min

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The disks were held between pin-point contacts in both nickel plating baths. The contacts were changed several times in each bath.

9. Rinse.

10. Vacuum heat-treatment at 600°C for 1/2 hour.

The conditions for vacuum heat-treatment were based on metallographic examination of a set of samples heat-treated at various times at 600 and 650°C. The apparent diffusion zone, produced by alloying of the nickel and serrulated uranium surface, was composed of a number of brittle intermetallic compounds. It was felt that such a diffusion layer should be kept thin in order to achieve good adherence of the nickel and uranium, and the vacuum heat treatment at 600°C for 1/2 hour appeared satisfactory and, in addition, provided a softening anneal for the nickel. Figures KS-995 and KS-996 show a sample prepared in the manner described, before and after heat-treatment.

Twenty-five samples plated with a total of one mil of nickel and heat-treated were corrosion tested in boiling water. The following is a summary of results:

% Samples	Time Period To Failure, hr
23	0 - 40
40	40 - 80
20	80 - 120
7	120 - 160
3	160 - 200
7	>200

An increase in the thickness of the nickel to about 2 mils on a series of six disks, followed by 600°C heat-treatment, raised the corrosion resistance in boiling water of all six to a period of 170 to 300 hours.

Refinements in the techniques used in the pretreatment and plating processes were developed on alpha-rolled uranium cylinders about 3/4 in. diameter x 1-1/2 in. long. However, it became apparent that preferential etching of the ends of the uranium cylinders by the anodic treatment was causing porosity in the plate. Corrosion generally started at points on the ends of the cylinders and resulted in undercutting and splitting of the nickel coating.

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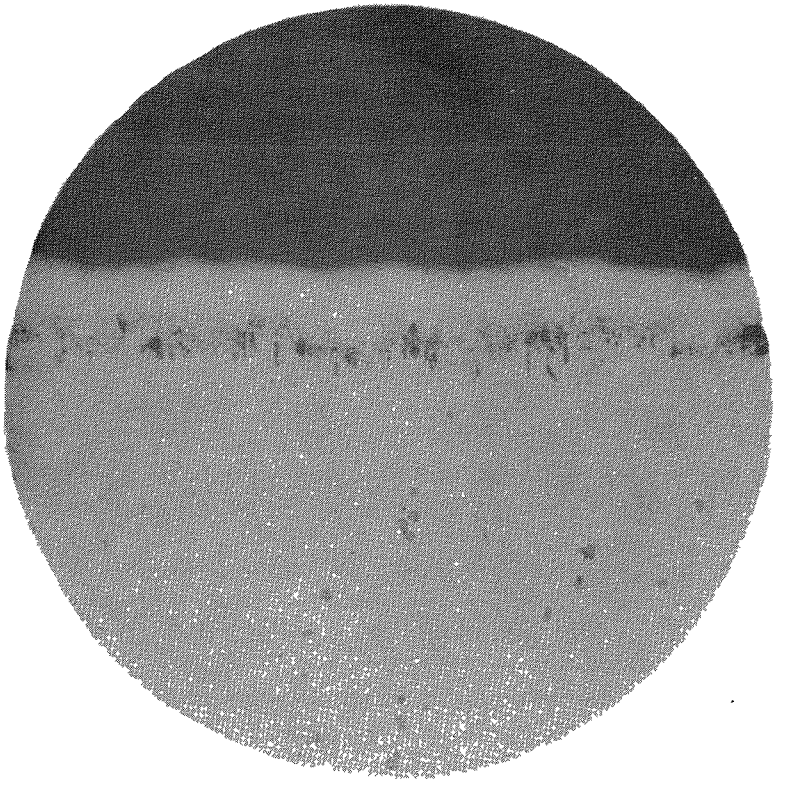

URANIUM TREATED ANODICALLY AND
AS NICKEL PLATED. 250X B. F.
AS POLISHED.

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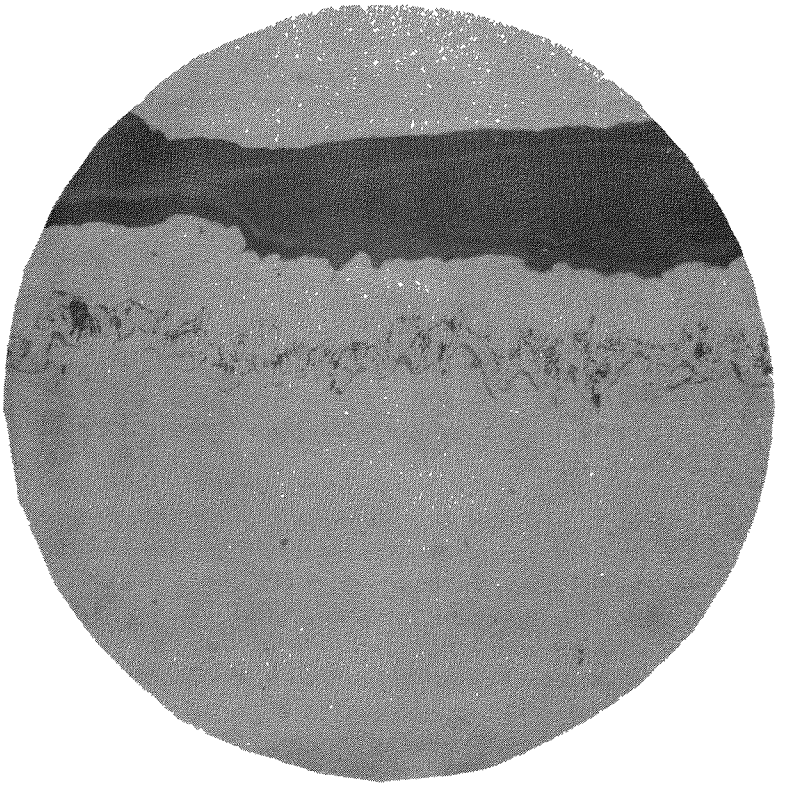

URANIUM TREATED ANODICALLY,
NICKEL PLATED, AND HEAT-TREATED
AT 600°C FOR 1/2 HR. 250X B. F.
AS POLISHED.

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This effect on cylindrical samples is evidenced in the following summary of the corrosion data on 25 alpha-rolled uranium cylinders plated with 2 mils of nickel and vacuum heat-treated at 600° for 1/2 hour:

% Samples	Time Period To Failure, hr
48	40 - 80
32	80 - 120
8	120 - 160
8	160 - 200
4	> 200

A fusion layer of tin was tried as a means of sealing the pores on the ends of the cylinders. Although this showed some promise, it was found difficult to prevent excessive alloying of the tin and nickel and also to keep the tin in a thin uniform sealing layer.

DEVELOPMENT OF AQUEOUS GRIT BLASTING AND CATHODIC PICKLING PRETREATMENT

To overcome the preferential etching by the anodic pretreatment, a new approach on pretreatment of the uranium was tried. This involved degreasing, blasting of the surface with an aqueous suspension of silica, and treating cathodically in an acid. The possibility existed that an activation of the uranium surface may occur during a cathodic treatment in acid if the following reaction involving atomic hydrogen should occur:

$$UO_2(\text{solid}) + 4H\ (\text{gas}) = U\ (\text{solid}) + 2H_2O\ (\text{liquid})$$

Thermodynamic data[8] indicate that this reaction could proceed, since the standard free energy change, ΔF°_{298} is -50.7 kcal/mole. Also, the cathodic treatment should provide an excellent scrubbing action on the surface of the uranium metal and therefore remove any mechanically entrapped foreign material.

Of a series of samples prepared using nitric, hydrochloric, phosphoric, and sulfuric acids for the cathodic treatment, those treated cathodically in sulfuric acid (35% by wt) were corrosion resistant for exceedingly long times (1000 hr). Because of the outstanding results of the samples prepared by use of a cathodic sulfuric acid treatment, subsequent work was done on electroplating alpha-rolled uranium cylinders using the following specific technique:

1. Vapor degrease.
2. Blast surface with aqueous suspension of 100 mesh SiO_2.
3. Rinse.
4. Cathodic treatment in 35% wt H_2SO_4 - H_2O for 30 sec at (25 amp/sq ft) room temperature.

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5. Rinse.

6. Nickel strike from "High Sulfate" bath for about 7 minutes. Pin-point contacts changed on sample at 5 minutes.

7. Rinse.

8. Build-up of nickel to about 2 mils in Watt's bath. Pin-point contacts changed 3 or 4 times.

9. Vacuum heat-treated at 600°C for 1/2 hour.

Figures KS-997, KS-998, and KS-999 show a sample as-plated and after heat-treatment.

Analysis of the results from over 50 samples showed that:

1. Of 37 samples corrosion tested in boiling water, the following occurred:

% Samples	Time Period To Failure, hr
14	20 - 40
10	40 - 80
10	80 - 120
6	120 - 160
0	160 - 200
60	> 200

2. Careful control of the "High Sulfate" and Watt's baths is necessary. The operating conditions of the "High Sulfate" bath should be: pH = 5.5, T = 45°C, 0.3 g/liter Duponol ME wetting agent. The operating conditions of the Watt's bath should be: pH = 4.0 to 4.2, T = 55°C, H_2O_2 as cathode depolarizer and anti-pitting agent. Continuous filtration and covers on the baths were necessary to eliminate dust.

3. The sources of failure in the samples that lasted less than 200 hours were pits in the plates due to incorrect plating conditions (eg. dust particles in bath), or fissures in the plates caused by the pin-point contacts used in this phase of the work.

4. The adhesion of as-plated nickel to alpha-rolled uranium was not as good as that obtained using an anodic pretreatment, but a vacuum heat-treatment at 600°C for 1/2 hour provided a thin (~0.0002 in.) uniform U-Ni diffusion layer. The strength of this diffusion layer has been measured as 6500 psi[9] average.

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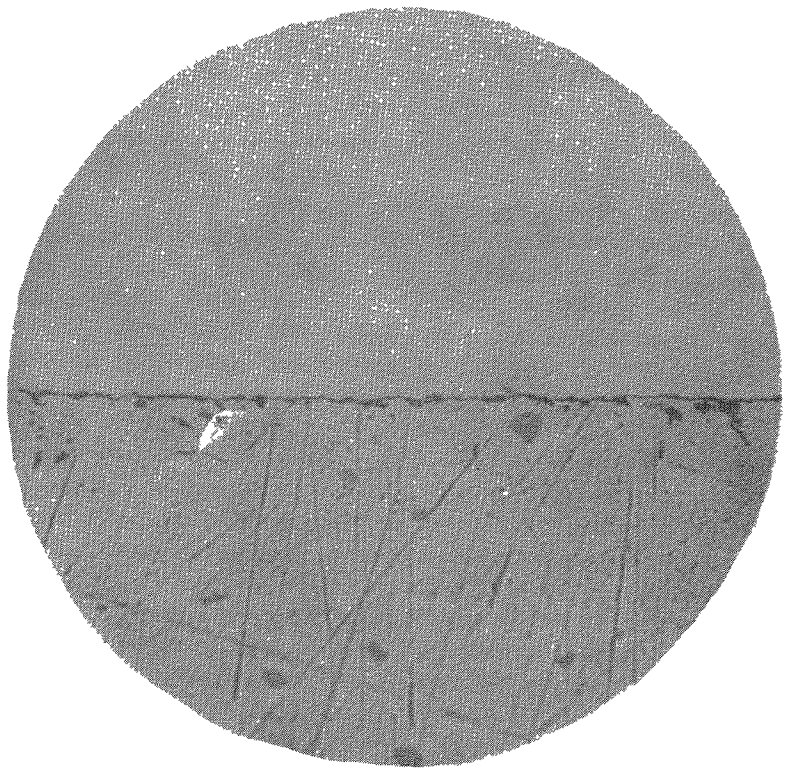

URANIUM TREATED CATHODICALLY
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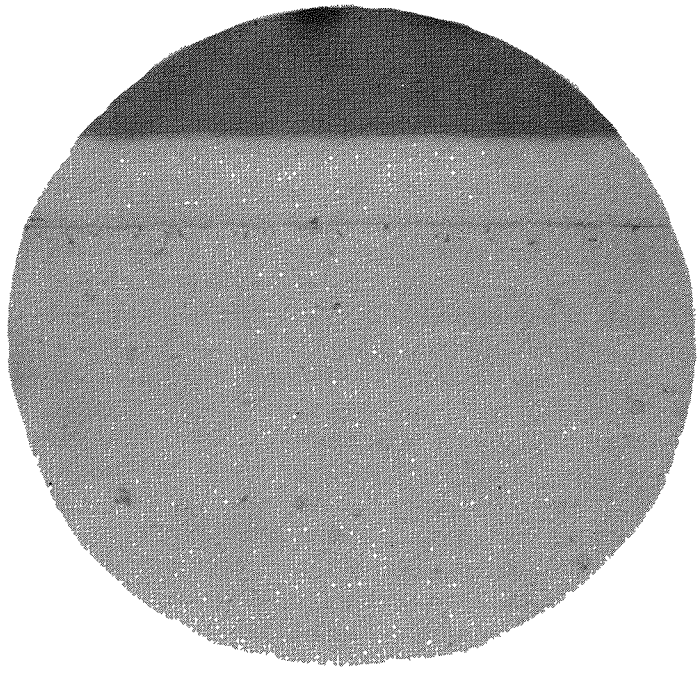

URANIUM TREATED CATHODICALLY,
NICKEL PLATED, AND HEAT-TREATED
AT 600°C FOR 1/2 HR. 250X B. F.
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URANIUM TREATED CATHODICALLY,
NICKEL PLATED, AND HEAT-TREATED
AT 600°C FOR 1/2 HR. 1000X B. F.
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APPLICATION TO HANFORD FUEL ELEMENTS

Facilities were set up to electroplate Hanford fuel elements (about 1-1/2 in. diameter x 4 in. long). (See Figure 1132817.) Two eight-gallon Koroseal lined steel tanks were used for the baths. Vitrosel immersion heaters, controlled by Fenwal units, were employed to maintain correct bath temperatures. Continuous filtration was provided by two Sethco filter pumps. Double layered nylon bags were used on the anodes. Plexiglas covers were installed over each tank to prevent dust in the air from settling on the surfaces of the electroplating baths (Figure 1132820), because dust particles can serve as sources of pitting in nickel plates. The baths were made using C.P. and analytical grades of low-cobalt nickel salts and were put through the conventional purification cycle[10] before use. The electroplating racks had two stainless steel pin-point contacts that engaged the cylindrical samples on each end (Figure KS-595).

On application of nickel electroplated coatings to a group of Hanford slugs by the previously described procedure, it was found that extensive blistering of nickel occurred upon vacuum heat-treatment at 600°C for 1/2 hour. This blistering seemed to occur mainly on the ends of the cylinders. An analysis of the problem showed:

1. The as-received Hanford slugs were made from alpha-rolled, beta-treated uranium, while the electroplating was developed mainly on small alpha-rolled cylinders and only a few beta-treated samples made at KAPL. (The KAPL beta heat-treatment was performed in molten tin-lead baths while Hanford used salt bath heating.)

2. Two pin contacts were used for the small and large cylinders. In the latter case the current densities around the ring points would be much greater and codeposition of hydrogen with the nickel may be excessive. The adsorption and occlusion of hydrogen could cause blistering.

3. The surface quality of the Hanford slugs left much to be desired. Longitudinal striations were present on most of them in varying degrees. These striations could entrap solutions which could cause blistering of the nickel coatings.

W. E. Ray[11] (Hanford Works, General Electric Co.) identified the gas causing blistering as mainly hydrogen and found that substantial amounts of this gas were introduced during the salt bath beta heat-treatment to the outer layers (0.20 in. thick) of alpha-rolled cylinders. At his suggestion, the Hanford slugs were outgassed at 600°C for 5 hours prior to pretreatment for electroplating.

Concurrently, work was being performed in this laboratory on modification of any part of our plating procedure that could cause this blistering. Re-

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duction of current densities used in the plating, introduction of a cradle-type plating rack with three contact points (Figure KS-596), together with the outgassing before plating, reduced blistering on the Hanford slugs substantially.

A study of the effect of uranium surface quality on the quality of the subsequent nickel coating was made on a random lot of 26 cylinders. Before plating, the cylinders were graded "good" (no surface defects), "fair" (minor scratches, striations and/or pits), and "poor" (deep striations and/or pits). After plating and heat-treating, no blistering occurred in the "good" slugs, 23% of the "fair" group blistered, and 63% of the ones rated "poor" blistered. This study showed definitely the direct relationship between uranium surface quality and blistering of the nickel plating after heat-treatment.

CONCLUSIONS

It is feasible to produce essentially nonporous electroplated nickel coatings (about 2 mils thick) on uranium. The best protection for a given thickness of nickel is obtained when applied to a relatively smooth uranium surface obtained by aqueous grit blasting and cathodic pickling in sulfuric acid. The "as-plated" adherence of this nickel plate is not as good as that obtained using an anodizing pretreatment on the uranium, but a diffusion bond is obtained on heat-treatment at 600°C for 1/2 hour. Careful control of uranium metal quality, such as gas content and surface imperfections, in addition to the various steps of the electroplating techniques, is imperative to achieve nonporous, diffusion-bonded nickel plates.

Because of the high absorption cross section of nickel, it is desirable to reduce the coating thickness and still retain its nonporous nature. Preliminary indications are that 1-1/2 mils, and possibly even 1 mil of nickel, applied by the present procedure, may be satisfactory. Further studies are needed to accomplish this reduction in thickness and also to reduce build-up of excess nickel at corners. Periodic reverse current plating is one approach that may be fruitful.

The introduction of hydrogen into uranium and the reduction of surface oxides by atomic hydrogen during cathodic treatment in acidic solutions deserve further study.

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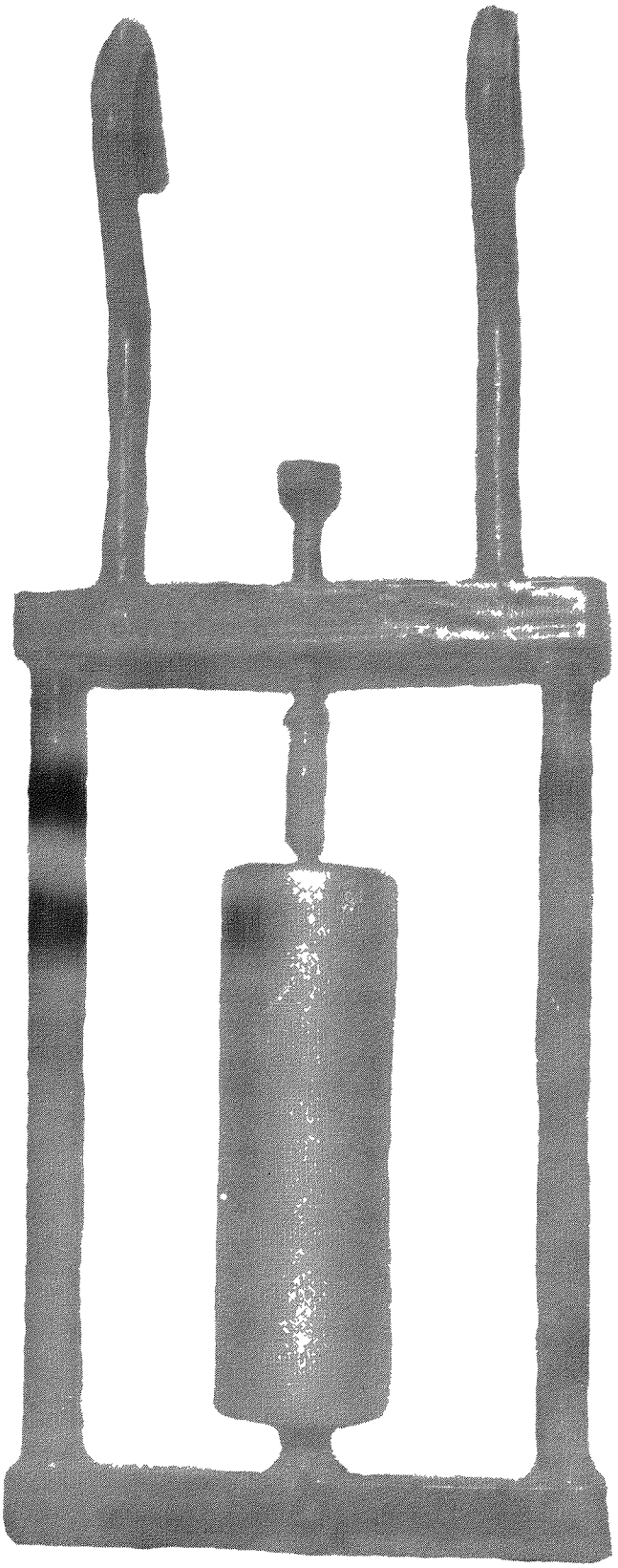

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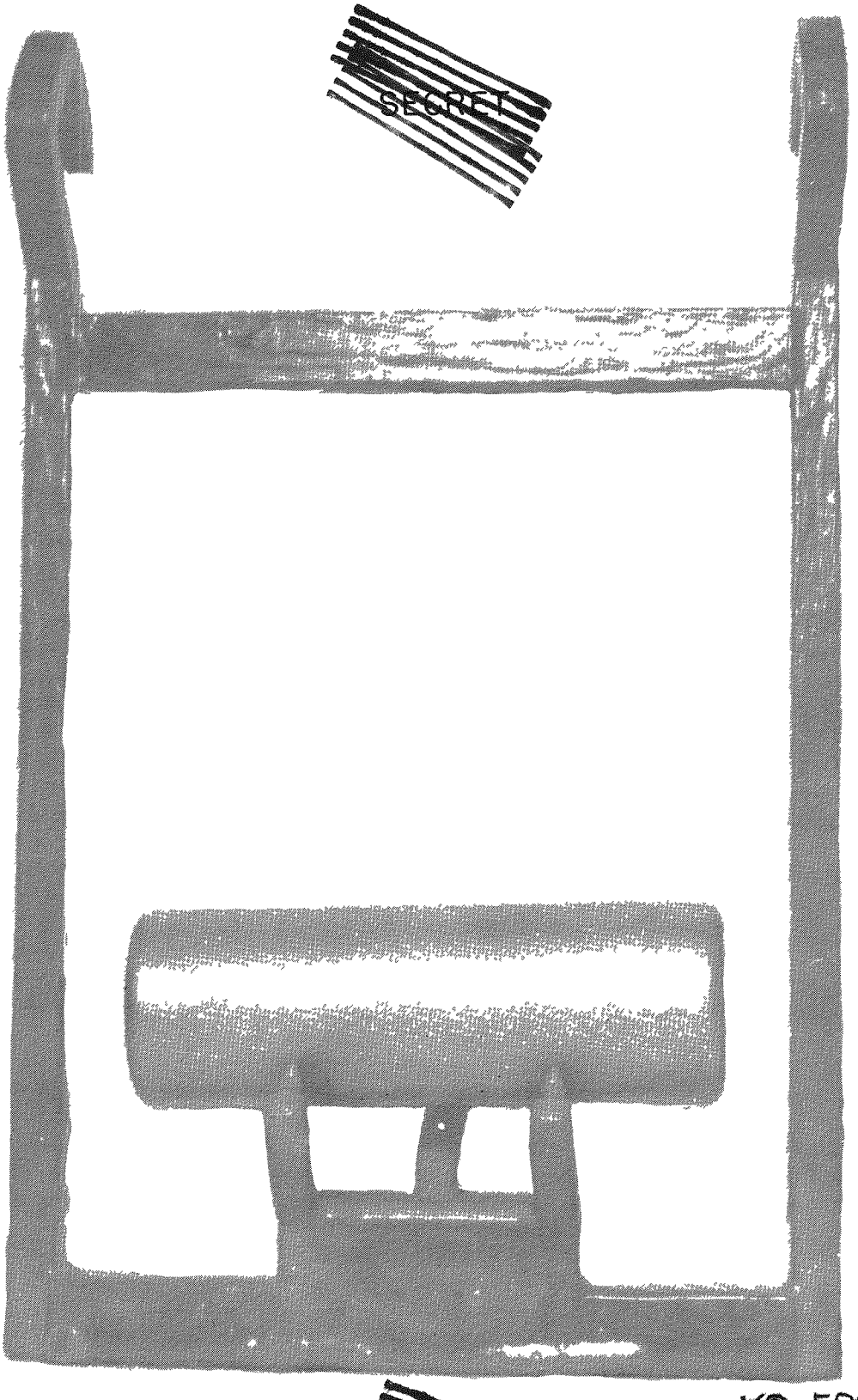

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